



# Preparation of organic/inorganic hybrid nanocomposites by ultraviolet irradiation and their packaging applications for organic optoelectronic devices

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## ABSTRACT

By ultraviolet (UV)-assisted synthetic procedure, we have successfully prepared several UV curable organic/inorganic hybrid nanocomposites with excellent gas barrier capabilities, moderate hardness, and good adhesive strength. The experimental results reveal that the physical properties of nanocomposites depend on their chemical structures. Therefore, introduction of silicone and polyurethane (PU) into the Acrylics backbone dramatically raises the adhesive strength as well as refractive index and lowers the gas penetration. Furthermore, we have also applied lab-made nanocomposite **g** for the encapsulation of organic optoelectronic devices such as OLEDs, flexible OLEDs, and organic solar cells. With the package of lab-made nanocomposite **g**, the organic optoelectronic devices effectively resist the entry of moisture and oxygen in the air, extending the lifetimes.

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## 1. Introduction

Organic/inorganic hybrid nanocomposites, composed of organic polymer matrices (e.g. acrylics, epoxy, and silicone) and inorganic nano-fillers (e.g. silica (SiO<sub>2</sub>), titanium oxide (TiO<sub>2</sub>), zinc oxide (ZnO), and alumina (Al<sub>2</sub>O<sub>3</sub>)) [1], have recently attracted much attention because of their miscellaneous applications such as fuel cells [2], nonlinear optics [3], lithium batteries [4], gas sensors [5], flame retarding [6], photochromism [7], and so on. Since they can resist the entry of moisture and oxygen in the air, they have also utilized in the preservation of beverages and food [8]. Although the literatures about the promotion of lifetimes for the encapsulation of electronic devices with metals or glass cans have been reported [9–11], the process of package is expensive and complicated. Nowadays, most optoelectronic devices are encapsulated with inorganic materials (e.g. SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, etc.) [12], polymers (e.g. polyacrylics, PET, poly(p-xylylene), etc.) [13,14] and their combinations (i.e. organic/inorganic hybrid composites) [15] owing to the advantage of smaller form factors, low cost, and improved manufacturability [16]. With the gas resistance of organic/inorganic hybrid nanocomposites, the lifetimes of optoelectronic devices can be effectively

prolonged because oxygen and moisture in the atmosphere cause the corrosion for organic layers, metal electrodes, and other materials of devices [17,18]. However, the conventional processes for the syntheses of organic/inorganic hybrid nanocomposites are time and energy-consuming.

In this paper, we have rapidly synthesized several organic/inorganic hybrid nanocomposites with ultraviolet (UV)-assisted polymerization and then investigated their physical properties. Because they possess excellent adhesive strength, moderate hardness, and good transparencies, we have also applied them for the package of organic optoelectronic devices such as organic light emitting diodes (OLEDs), flexible OLEDs, and organic solar cells. The experimental results manifest that the lifetimes of organic optoelectronic devices dramatically raise and lab-made organic/inorganic hybrid nanocomposites exhibit excellent gas barrier capability.

## 2. Experimental

### 2.1. Materials

All of monomers (Fig. 1), photoinitiators (Fig. 2), organic materials for electronic devices (Fig. 3), solvents, and fillers (silica; 30–100 nm) used in the experiment were purchased from Aldrich Co. and used without further purification.

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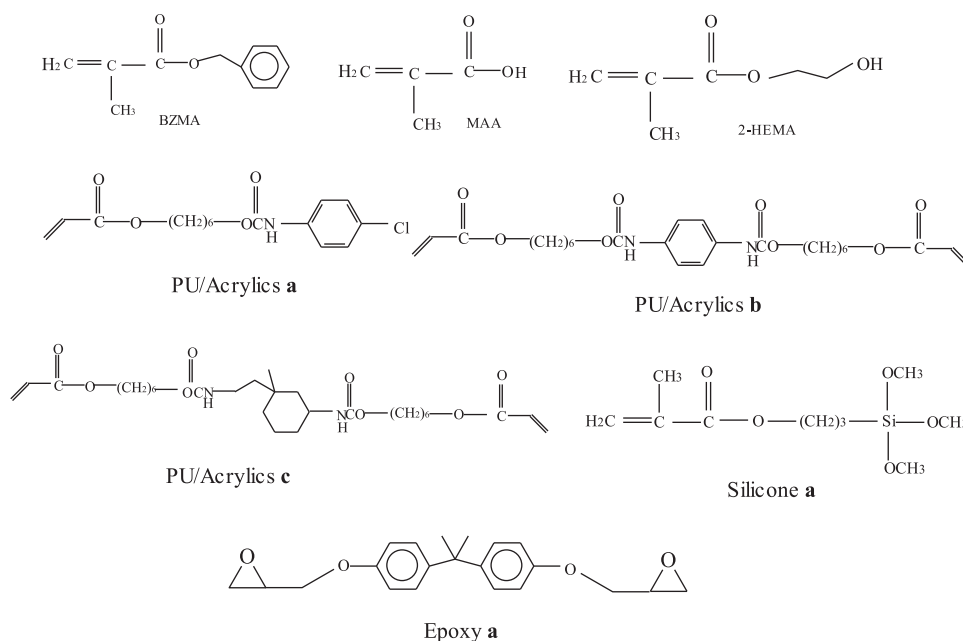


Fig. 1. Monomers for lab-made organic/inorganic hybrid nanocomposites.

## 2.2. Preparation of UV-curable organic/inorganic hybrid nanocomposites

### 2.2.1. Preparation of nanocomposite **a** (Scheme 1)

Benzyl methacrylate (BZMA; 117 g), methyl methacrylate (MAA; 86 g), 2-hydroxyethyl methacrylate (2-HEMA; 130 g), silica (39 g), and I-184 (6 g) were mechanically stirred and irradiated by a UV lamp (Entela UVP; 100 W) at room temperature for 20 min. Then nanocomposite **a** was obtained. The data for weight-average molecular weight ( $M_w$ ), number-average molecular weight ( $M_n$ ),  $M_w/M_n$  ratio, and viscosity of nanocomposite **a** were tabulated in Table 1.

### 2.2.2. Preparation of nanocomposite **b**, **c**, and **d** (Schemes 2–4)

PU/Acrylics **a** (333 g), silica (39 g), I-184 (2 g), and I-369 (4 g) were mechanically stirred and irradiated by a UV lamp (Entela

UVP; 100 W) at room temperature for 20 min. Then nanocomposite **b** was obtained. With the same amounts of monomers, fillers, and photoinitiators, nanocomposite **c** and **d** were synthesized by the similar procedure. In addition, their viscosities and molecular weights were tabulated in Table 1.

### 2.2.3. Preparation of nanocomposite **e** (Scheme 5)

Silicone **a** (333 g), silica (39 g), I-184 (3 g), I-369 (1.5 g), and I-ITX (1.5 g) were mechanically stirred and irradiated by a UV lamp (Entela UVP; 100 W) at room temperature for 20 min. Then nanocomposite **e** was obtained. As shown in Table 1, its viscosity and molecular weight were described.

### 2.2.4. Preparation of nanocomposite **f** (Scheme 6)

Silicone **a** (166.5 g), 2-HEMA (166.5 g), silica (39 g), I-184 (3 g), I-369 (1.5 g), and I-ITX (1.5 g) were mechanically stirred and irradiated by a UV lamp (Entela UVP; 100 W) at room temperature for 20 min. Then nanocomposite **f** was obtained. Moreover, its viscosity and molecular weight were listed in Table 1.

### 2.2.5. Preparation of nanocomposite **g** (Scheme 7)

Silicone **a** (166.5 g), PU/Acrylics **b** (166.5 g), silica (39 g), I-184 (2.7 g), I-369 (1.35 g), I-ITX (1.35 g), and I-127 (0.6 g) were mechanically stirred and irradiated by a UV lamp (Entela UVP; 100 W) at room temperature for 20 min. Then nanocomposite **g** was obtained. As manifested in Table 1, its molecular weight and viscosity were described.

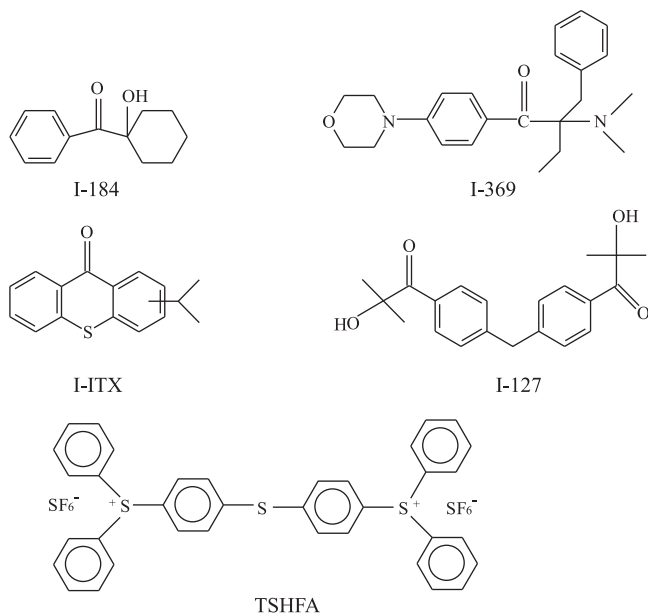


Fig. 2. Photoinitiators for lab-made organic/inorganic hybrid nanocomposites.

Table 1

The data for viscosity and molecular weight of lab-made organic/inorganic hybrid nanocomposites.

Nanocomposite	Viscosity (cps)	$M_w$	$M_n$	$M_w/M_n$
<b>a</b>	15,100	233,100	113,300	2.06
<b>b</b>	8600	123,100	66,100	1.86
<b>c</b>	23,200	275,000	138,400	1.99
<b>d</b>	22,800	262,000	125,400	2.09
<b>e</b>	6900	73,700	43,200	1.71
<b>f</b>	8800	103,100	56,700	1.82
<b>g</b>	12,400	118,600	62,800	1.89
<b>h</b>	10,700	93,200	48,600	1.92

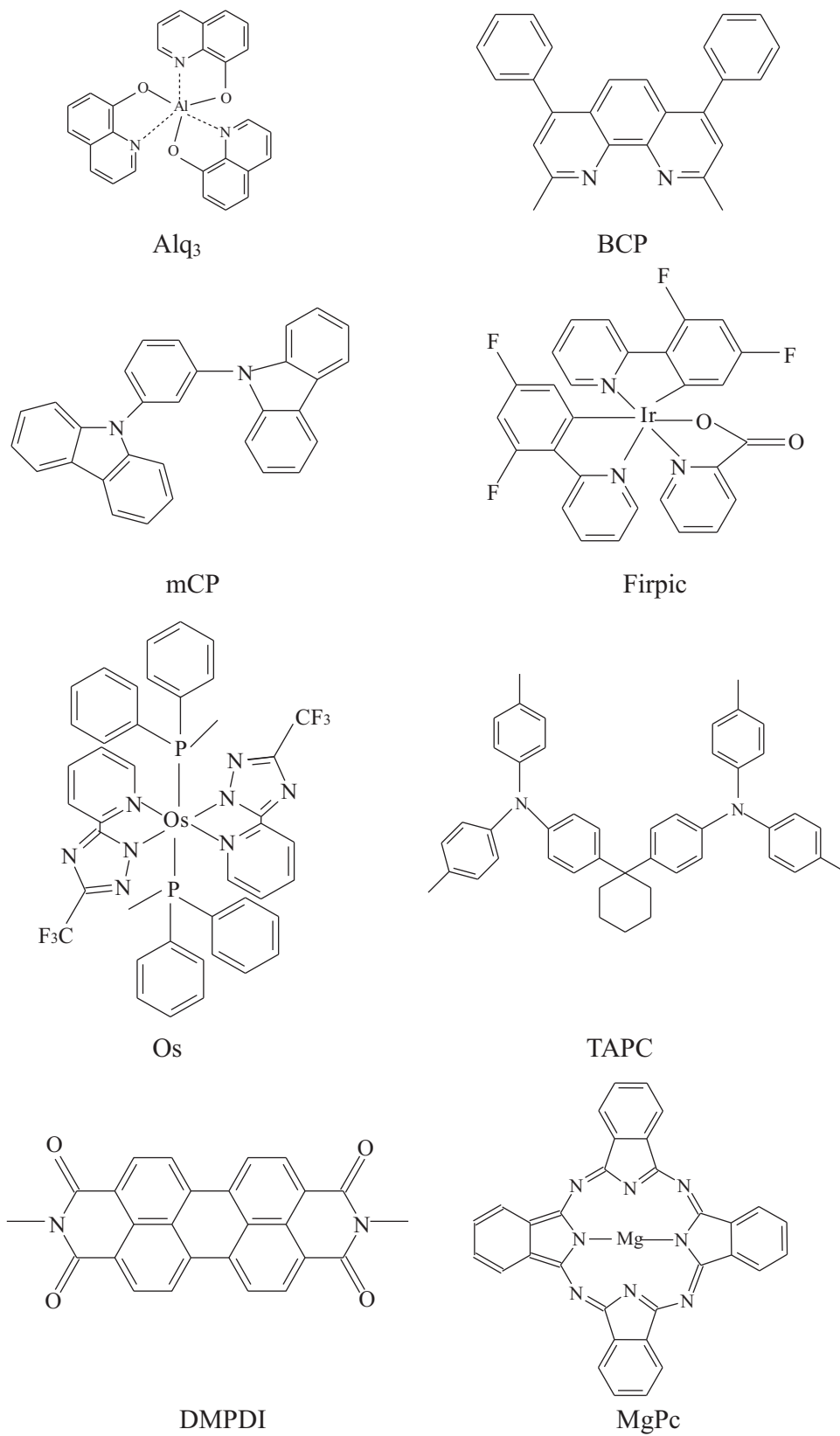
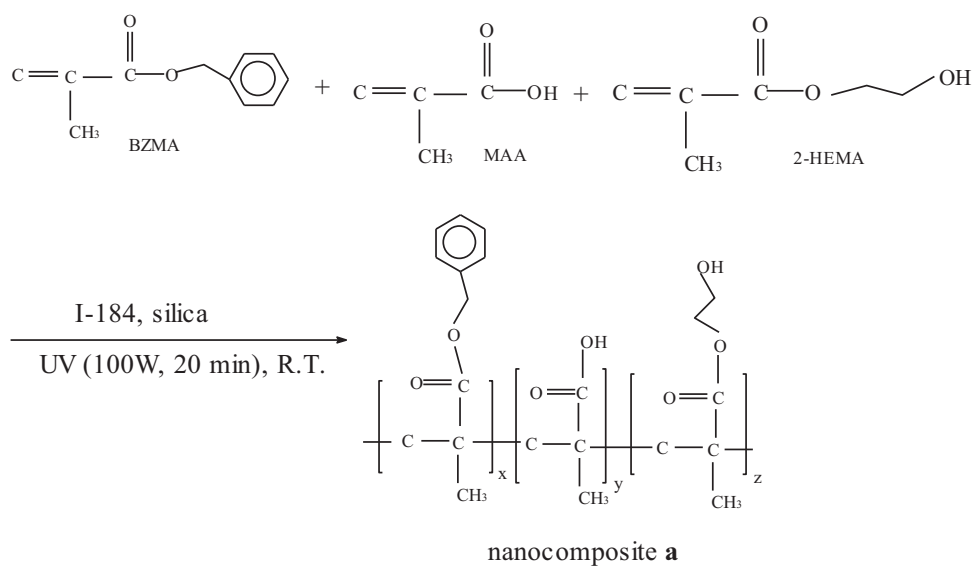
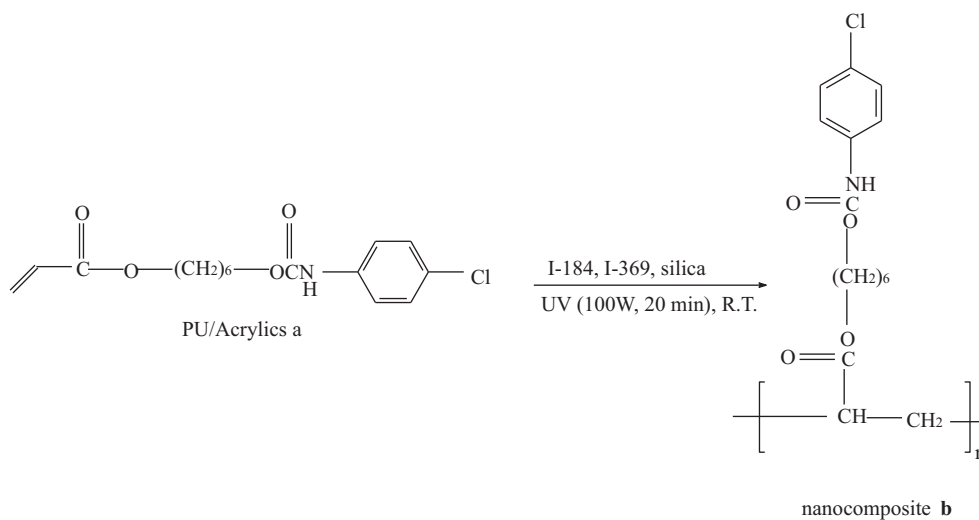
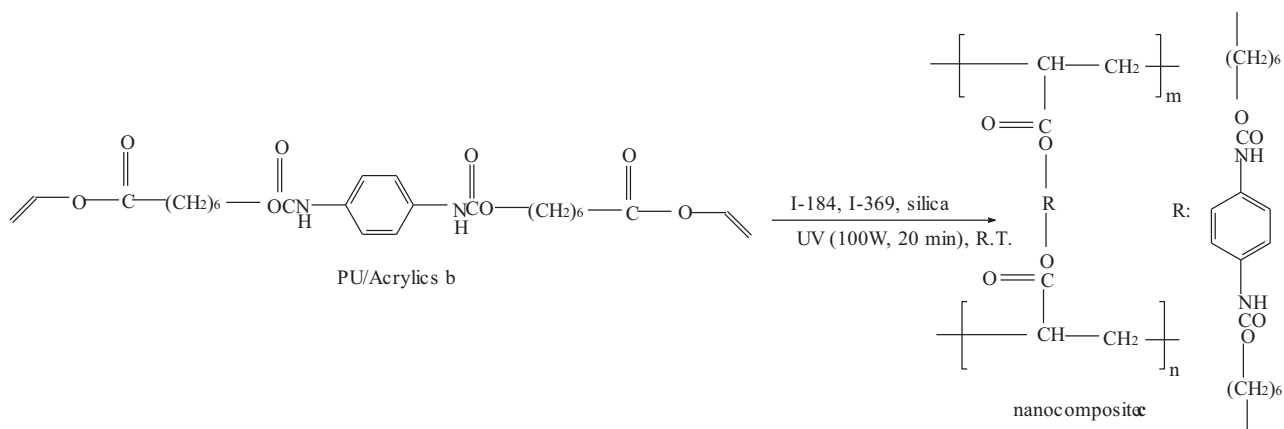
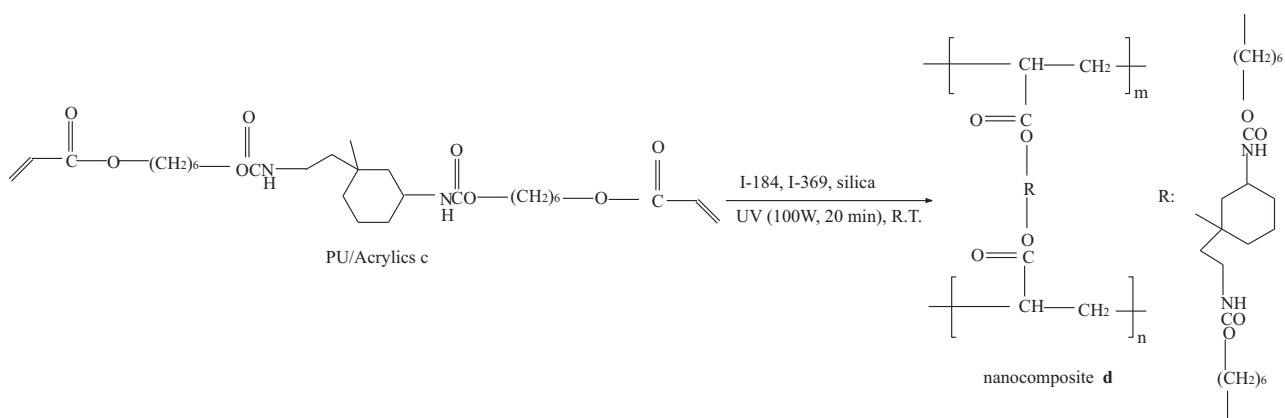
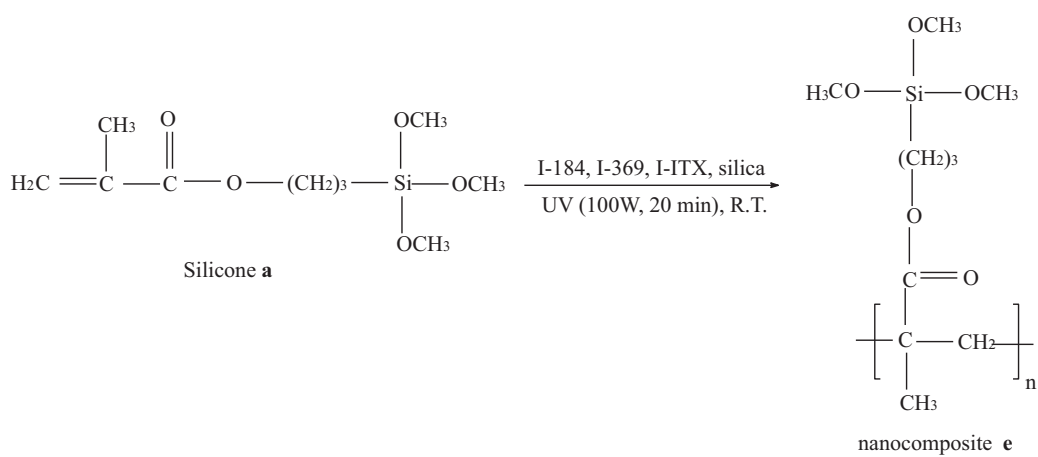


Fig. 3. Organic materials for optoelectronic devices.

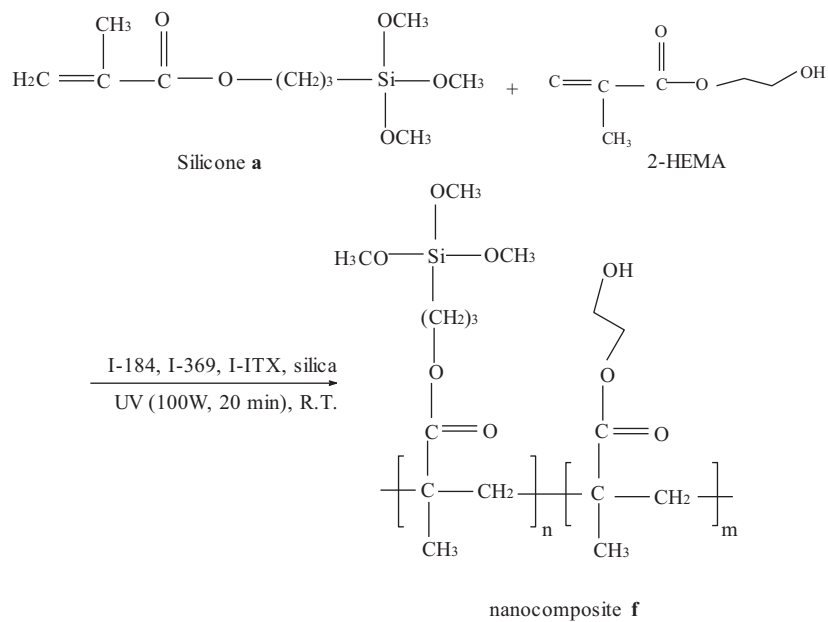
**Scheme 1.** Synthesis of nanocomposite a.**Scheme 2.** Synthesis of nanocomposite b.**Scheme 3.** Synthesis of nanocomposite c.



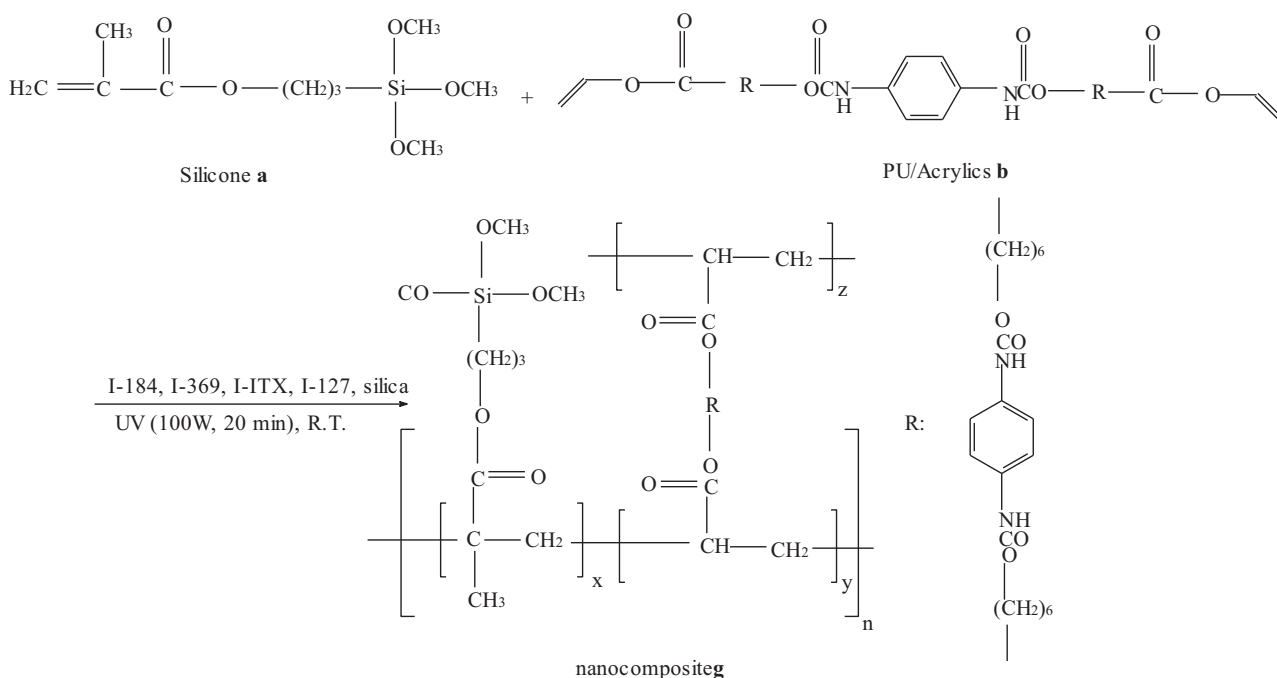
Scheme 4. Synthesis of nanocomposite d.



Scheme 5. Synthesis of nanocomposite e.



Scheme 6. Synthesis of nanocomposite f.

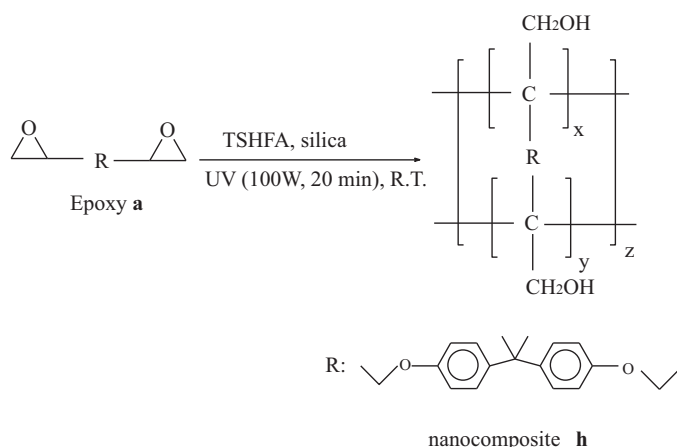
Scheme 7. Synthesis of nanocomposite **g**.

### 2.2.6. Preparation of nanocomposite **h** (Scheme 8)

Epoxy **a** (333 g), alumina (39 g), and TSHFA (6 g) were mechanically stirred and irradiated in a UV lamp (Entela UVP; 100 W) at room temperature for 20 min. Then nanocomposite **h** was obtained. The data for  $M_w$ ,  $M_n$ ,  $M_w/M_n$  ratio, and viscosity of nanocomposite **h** were tabulated in Table 1.

### 2.3. Instruments

Molecular weights and viscosities were measured by a Waters Alliance GPC V2000 and a Viscolite 700, respectively. Furthermore, we examined the adhesion strength, hardness, transparencies, refractive indices, and gas permeation rates with a micro-computer universal testing machine (Hung Ta Co.), a pencils type film hardness tester (ZSH 2090), a HITACHI U-3300, a Filmetrics F20, and an Illinois-8501, respectively. The UV lamps used for syntheses and curing were respectively Entela UVP 100 W and 2450 W. Moreover, we recorded the photoelectric properties and lifetimes of OLEDs by Keithley 2400 and Spectrascan PR650, respectively. The film thick-

Scheme 8. Synthesis of nanocomposite **h**.

ness was measured by a surface profiler (TENCOR P-10). In addition, the current–voltage ( $I$ – $V$ ) curves for organic solar cells were measured in the air by an instrument (Keithley 238), whose accuracy can reach picoampere, under illumination of white light from a 300 W halogen lamp (Saturn Co.) whose intensity was recorded on a radiometer (IL-1700). The lifetimes for OLEDs, flexible OLEDs, and organic solar cells were examined in real-time conditions.

### 2.4. Fabrication of OLEDs and flexible OLEDs

The fabrication of OLEDs was executed in a glove box. The indium tin oxide (ITO) glass ( $5 \Omega/\text{square}$ ) was ultrasonically washed with the acetone, methanol, and de-ionized water for 5 min. After dried with a stream of nitrogen as well as the oven and treatment of  $O_2$  plasma for 90 s, we deposited 1,1-bis(4'-bis(4''-menthylphenyl)aminophenyl)cyclohexane (TAPC; hole transport layer; 40 nm), 9H-carbazole-9,9'-(1,3-phenylene)-bis-(9C1) (mCP; green emitting material)/Osmium(II) bis(3-(trifluoromethyl)-5-(4-tert-butylpyridyl)-1,2,4-triazolate) diphenylmethylphosphine (Os; red emitting material)/bis(3,5-difluoro-2-(2-pyridyl)phenyl-(2-carboxypyridyl)iridium III (Firpic; blue emitting material) mixture (25 nm; weight ratio = 82/17/1), 2,9-dimethyl-4,7-dimethylphenanthroline (BCP; electron transport layer; 15 nm), tris-[8-hydroxyquinoline]aluminium ( $Alq_3$ ; electron transport layer; 30 nm), lithium fluoride (LiF; electron injection layer; 0.5 nm), and aluminium (Al; cathode; 150 nm) layer by layer onto the ITO glass by vacuum evaporation. Finally, lab-made nanocomposite **g** (100  $\mu\text{m}$ ) was deposited on the Al electrode by spin-coating technique (stage I: 1500 r.p.m. for 20 s; stage II: 3500 r.p.m. for 30 s) and cured by UV illumination for 10 s. (Fig. 4(a)). The similar process was executed in the fabrication of flexible OLEDs except the ITO glass was replaced with the ITO PET (poly(ethylene terephthalate)) (Fig. 4(b)).

### 2.5. Fabrication of organic solar cells

The fabrication of organic solar cells was also executed in a glove box. The ITO glass ( $5 \Omega/\text{square}$ ) was ultrasonically washed

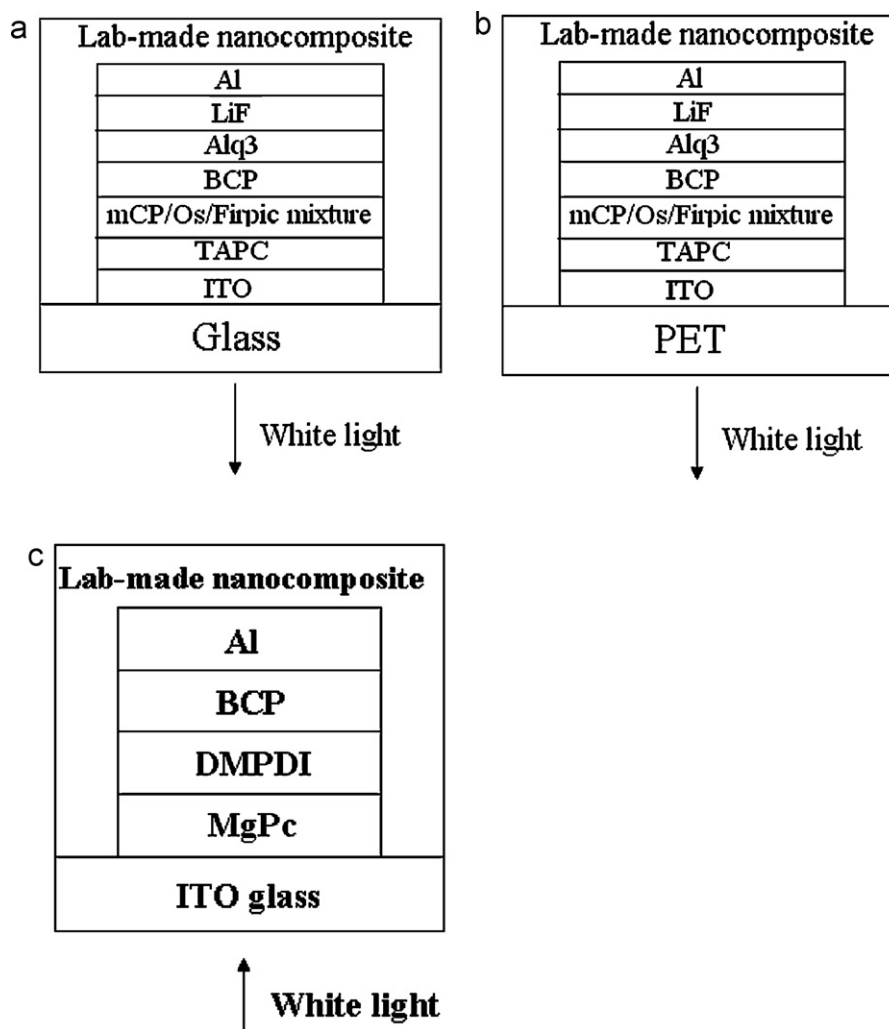


Fig. 4. Structures of lab-made (a) OLEDs, (b) flexible OLEDs, and (c) organic solar cells.

with the acetone, methanol, and de-ionized water for 5 min, dried with a stream of nitrogen as well as the oven, and treated with  $O_2$  plasma for 90 s. Then we deposited magnesium phthalocyanine (MgPc; p-type semiconductor; 30 nm), N,N'-bis(1,5-dimethyl)-3,4:9,10-perylenebis(dicarboximide) (DMPDI; n-type semiconductor; 50 nm), 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP; electron transport layer; 15 nm), and aluminium (Al; anode; 90 nm) layer by layer onto the ITO glass with vacuum evaporation. Finally, lab-made nanocomposite **g** (100  $\mu\text{m}$ ) was deposited on the Al electrode by spin-coating technique (stage I: 1500 r.p.m. for 20 s; stage II: 3500 r.p.m. for 30 s) and cured by UV illumination for 10 s (Fig. 4(c)).

### 3. Results and discussion

#### 3.1. Preparation of organic/inorganic hybrid nanocomposites by UV-assisted polymerization

UV, which is the electromagnetic radiation of 180–400 nm, exhibits high energy to cause the dissociation of photoinitiators, consequently producing free radical or cationic polymerization [19]. In this study, all the synthetic reactions with UV procedure take only 20 min and proceed without solvents as manifested in Schemes 1–8 while those with conventional thermal methods take 8–24 h and proceed with solvents. In addition, the curing time for

**Table 2**  
The physical properties of lab-made organic/inorganic hybrid nanocomposites.

Nanocomposite	Adhesive strength (kg/cm)	Hardness	Transparency (%)	Permeation rate for oxygen ( $\text{g}/\text{m}^2$ day)	Permeation rate for moisture ( $\text{g}/\text{m}^2$ day)
<b>a</b>	0.3	2H	90	0.25	0.21
<b>b</b>	0.8	2H	87	0.20	0.17
<b>c</b>	1.4	3H	90	0.10	0.08
<b>d</b>	1.3	H	91	0.14	0.11
<b>e</b>	1.6	3B	95	0.08	0.06
<b>f</b>	1.7	2B	93	0.07	0.05
<b>g</b>	2.1	B	92	0.05	0.03
<b>h</b>	1.5	3H	46	0.17	0.13

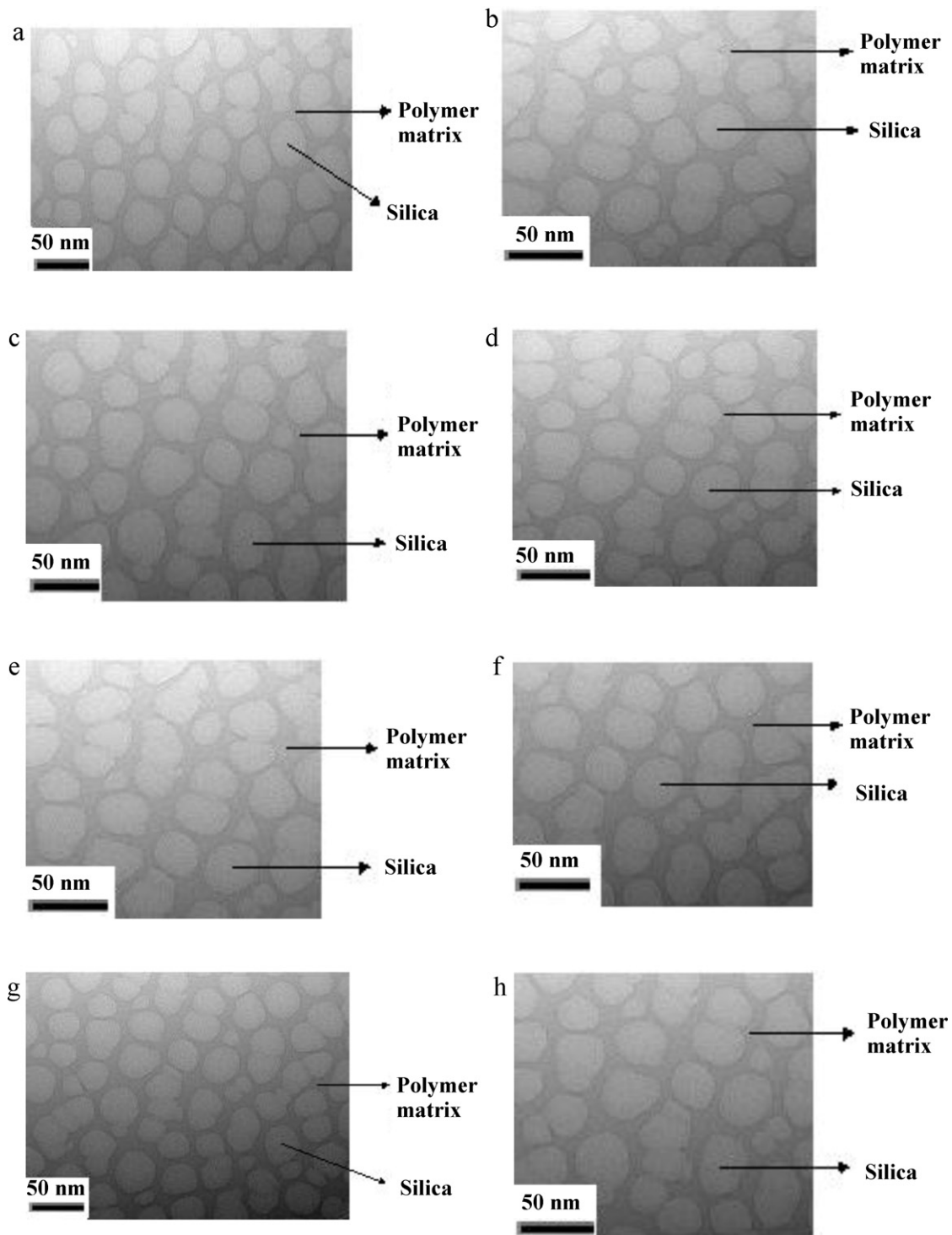


Fig. 5. TEMs of lab-made nanocomposite (a) a, (b) b, (c) c, (d) d, (e) e, (f) f, (g) g and (h) h.

lab-made organic/inorganic hybrid nanocomposites under UV irradiation is only 10 s while that with conventional thermal method is several hours. With UV procedure, the whole production efficiency can be increased and the product is immediately ready for testing, shipment, and storage rather than a multi-step thermal drying process. Furthermore, UV process has lower energy consumption and is also an environmentally friendly technology without emissions of volatile organic compounds (VOCs) and flammability. These merits fit the demands for the clean and low-cost preparation of organic/inorganic hybrid nanocomposites.

### 3.2. Physical properties of lab-made organic/inorganic hybrid nanocomposites

As shown in Table 2, the physical properties of lab-made organic/inorganic hybrid nanocomposites deeply depend on the chemical structures of organic polymers since the amounts of fillers, initiators, and photoinitiators utilized for the preparation of organic polymer/filler hybrids are the same. In case of simple acrylics nanocomposite (i.e. nanocomposite a), its adhesive strength, hardness, transparencies, permeation rate for oxygen, and



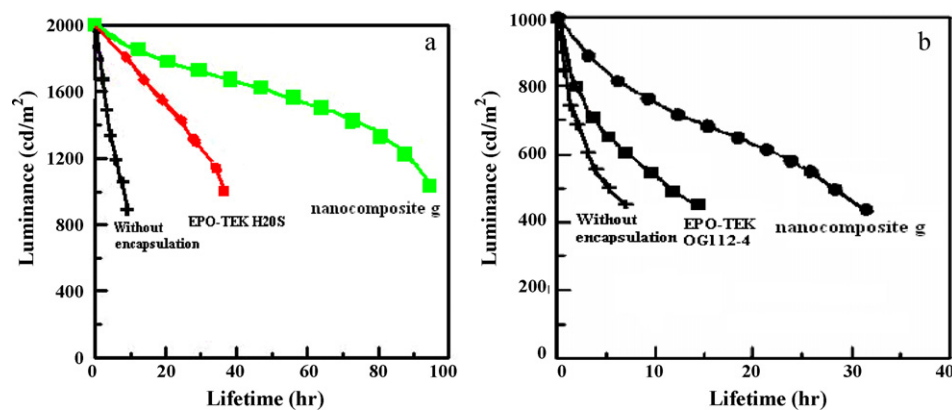


Fig. 6. Lifetimes of lab-made (a) OLEDs actuated at 6 V and (b) flexible OLEDs actuated at 6 V.

permeation rate for moisture are 0.3 kg/cm, 2H, 90%, 0.25 g/m<sup>2</sup> day, and 0.21 g/m<sup>2</sup> day, respectively. When PU and silicone are introduced into the backbone of organic/inorganic hybrid nanocomposite, nevertheless, its adhesive strength and refractive index dramatically raise and the gas penetration drops. This result comes from that PU and silicone exhibit higher surface energy than acrylics [20]. Therefore, introduction of PU and silicone causes the increase of adhesive strength to the glass, also enhancing gas blocking capability.

Among PU/Acrylics nanocomposites (i.e. nanocomposite **b**, **c**, and **d**), nanocomposite **c** exhibits the largest adhesive strength (1.4 kg/cm) and lowest gas penetration (oxygen: 0.10 g/m<sup>2</sup> day; moisture: 0.08 g/m<sup>2</sup> day). According to the experimental results, we also find that the hardness of PU/Acrylics nanocomposites with phenyl rings (i.e. nanocomposite **b** and **c**) is higher than that with cycloalkyl rings (i.e. nanocomposite **d**), revealing the hardness of PU/Acrylics nanocomposites extremely depends on their chemical structures. Moreover, the hardness of PU/Acrylics nanocomposites with two functional groups (i.e. nanocomposite **c**) is higher than that with one functional group (i.e. nanocomposite **b**).

In case of Silicone/Acrylics nanocomposites (i.e. nanocomposite **e** and **f**), their adhesive strength, refractive indices, and gas penetration are obviously superior to those of simple acrylics nanocomposite (i.e. nanocomposite **a**). Among them, the adhesive strength of nanocomposite **f** can reach 1.7 kg/cm and its permeation rates for oxygen and moisture are 0.07 and 0.05 g/m<sup>2</sup> day, respectively. Nonetheless, introduction of silicone drastically decreases the hardness of nanocomposites so that the hardness of nanocomposites **e** and **f** becomes 3B and 2B, respectively.

In order to further improve the physical properties of lab-made organic/inorganic hybrid nanocomposites, we have tried to add both PU and silicone into the backbones of acrylics to form nanocomposite **g**, whose adhesive strength, hardness, permeation rate for oxygen, and permeation rate for moisture are 2.1 kg/cm, B, 0.05 g/m<sup>2</sup> day, and 0.03 g/m<sup>2</sup> day, respectively. The experimental

results manifest that introduction of PU and silicone substantially improves the adhesive strength and gas penetration.

Although we have also successfully synthesized epoxy nanocomposite (i.e. nanocomposite **h**) with good adhesive strength (1.5 kg/cm) and low gas penetration (oxygen: 0.17 g/m<sup>2</sup> day; moisture: 0.13 g/m<sup>2</sup> day), color stain takes place during UV synthetic and curing process, leading to their poor transparencies (46%). Moreover, nano-fillers (i.e. silica) were homogeneously dispersed in the polymer matrices as shown in Fig. 5(a)–(h), indicating that the prepared materials were nanocomposites.

Among all the lab-made nanocomposites, nanocomposite **g** possesses the best physical properties compared with other material groups because their polymer matrices (i.e. Silicone/PU/Acrylics) may have largest surface energy [20], causing the raise of adhesive strength to the glass and the increase of gas resistance. Since nanocomposite **g** exhibits fast curing duration (10 s), excellent adhesive strength (2.1 kg/cm), moderate hardness (H), and low gas penetration (oxygen: 0.05 g/m<sup>2</sup> day; moisture: 0.03 g/m<sup>2</sup> day), we have executed the encapsulation of organic optoelectronic devices (i.e. OLEDs, flexible OLEDs, and organic solar cells) with them to evaluate their gas barrier capability and to promote the lifetimes of organic optoelectronic devices.

### 3.3. Package of OLEDs and flexible OLEDs

As shown in Fig. 6(a), the luminance of OLEDs without encapsulation sharply drops while the device is actuated at 6 V and their half-lifetimes, defined as the duration when the luminance decays from the original amount to its half, are only 10 h. This result reveals that the oxygen and moisture in the atmosphere induces the erosion of metal electrode and organic layers. However, the half-lifetimes drastically rise to 95 h while nanocomposite **g** is packaged in the device. This result manifests that lab-made organic/inorganic hybrid nanocomposites can block the entry of moisture and oxygen in the air into the OLEDs, therefore quenching the degradation

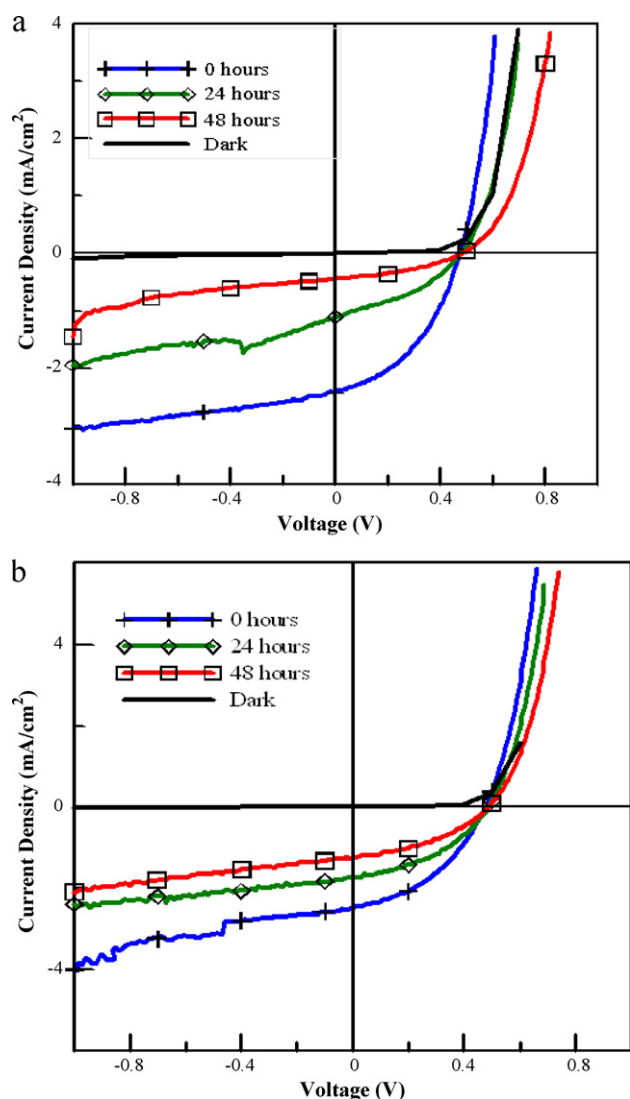
Table 3  
The photoelectric conversion properties of lab-made organic solar cells.

Encapsulating material	Actuating time (h)	Voc <sup>a</sup> (V)	Isc <sup>b</sup> (mA/cm <sup>2</sup> )	Fill Factor (%)	Efficiency (%)	Decay ratio (DR) <sup>c</sup> (%)
No encapsulation	0	0.48	2.46	40.2	0.487	–
	24	0.48	1.15	36.2	0.198	59.3
	48	0.48	0.48	36.9	0.092	81.1
Nanocomposite <b>g</b>	0	0.48	2.48	41.3	0.491	–
	24	0.48	1.75	42.6	0.377	23.2
	48	0.48	1.28	40.3	0.251	48.9

<sup>a</sup> Open circuit voltage.

<sup>b</sup> Short circuit current.

<sup>c</sup> Decay ratio is defined as  $DR = ((Eff_o - Eff_e)/Eff_o) \times 100\%$  where  $Eff_o$  and  $Eff_e$  represent the efficiency of organic solar cell for original state (actuating time = 0 h) and experimental state (actuating time = 24 or 48 h), respectively.



**Fig. 7.** *I*-*V* curves of lab-made organic solar cells ((a): without encapsulation and (b): with lab-made nanocomposite **g**).

of metal electrode as well as organic materials and extending the lifetimes. Compared with commercial UV curable encapsulating adhesives (EPO-TEK H20S; Epoxy technology Inc.), the devices with nanocomposite **g** have longer lifetimes and shorter curing time because the curing time and half-lifetimes of OLEDs with EPO-TEK H20S are 3 min and 38 h, respectively.

The similar result can be observed in case of flexible OLEDs as shown in Fig. 6(b). The half-lifetimes of flexible OLEDs with the encapsulation of nanocomposite **g** are 30 h, which are 4.3- and 2.2-folds longer than those without encapsulation and with EPO-TEK H20S, respectively, proving that lab-made organic/inorganic hybrid nanocomposites exhibit excellent gas barrier capability and are good encapsulating materials for flexible OLEDs.

### 3.4. Package of organic solar cells

The conversion of sunlight into electrical energy has been currently an essential issue because sunlight is an inexhaustible, clean, and environmentally friendly energy source. Although silicon-based solar cells exhibit good efficiencies, their costs are very high since purified silicon is expensive and the manufacturing procedure is complicated. Consequently, organic solar cells have been

alternative candidates for photoelectric conversion due to low cost and high processability. However, the lifetimes of organic solar cells have been a vital obstacle for the commercialization because oxygen and moisture in the air corrode the organic materials and metal electrodes of devices, highly reducing the lifetimes. Furthermore, most researches focus on the improvement of the efficiencies for solar cells but the studies about the promotion of lifetimes are less reported.

As shown in Fig. 7(a) and Table 3, lab-made organic solar cell without encapsulation exhibits rectifying effect under dark and photoelectric conversion properties under illumination of white light (conversion efficiency = 0.487%) because MgPc and DMPDI are respectively p-type and n-type semiconductors and thus p/n heterojunction generates. While the actuating time prolongs, however, the photoelectric conversion capability gradually drops since the moisture and oxygen in the atmosphere penetrate into the device, causing the erosion of organic materials and metal electrodes. After actuated for 48 h, the efficiency only remains to be 0.092% (decay ratio = 81.1%).

In order to further increase the lifetimes of organic solar cells, we have utilized nanocomposite **g** for the encapsulation of the devices. With nanocomposite **g**, as manifested in Fig. 7(b) and Table 3, the decay ratio dramatically reduces and can be only respectively 23.2 and 48.9% when the actuating time are respectively 24 and 48 h. This result demonstrates that nanocomposite **g** can prevent the entry of oxygen and moisture in the atmosphere into the device, successfully improving the lifetimes of organic solar cells.

## 4. Conclusions

We conclude that UV curable organic/inorganic hybrid nanocomposites with good adhesion strength, fast curing speed, moderate hardness, and excellent gas barrier capability have been successfully synthesized under UV irradiation. With the encapsulation of nanocomposite **g**, the lifetimes of OLEDs, flexible OLEDs, and organic solar cells can be increased drastically owing to their gas blocking effect.

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